Material Characterization Techniques

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Abstract—this review describes the Auger Electron Spectroscopy (AES), Low Energy Electron Diffraction (LEED), Raman Spectroscopy & Field Ion Microscopy (FIM) techniques for materials characterization at the atomic and molecular level along with composition, and vibration frequencies of a substance. The goal is to provide an overview of the utilization of these instrumental methods for analyzing the surfaces of material. Different spectroscopic techniques operate over different frequency ranges within this wide spectrum, depending on the processes and magnitudes of the energy changes. An introduction is presented on the basic principle behind the technique, the operation of the instrument, and the application of the technique.

Index Terms—Auger Electron Spectroscopy (AES), Low Energy Electron Diffraction (LEED), Raman Spectroscopy, Field Ion Microscopy (FIM)

I. INTRODUCTION

Spectroscopy is the study of the interaction of electromagnetic radiation with matter. It is an important tool that can find the molecular structures, composition, and vibration frequencies of a substance [1]. It is also be used to find the concentrations of reactants as functions of time in order to find the reaction intermediates. The collection of measurements absorbance of the compound as a function of electromagnetic radiation is called a spectrum. We can classify spectroscopic methods according to the region of the electromagnetic spectrum involved in the measurement. The regions include γ-ray, X-ray, ultraviolet (UV), visible, infrared (IR), microwave, and radio frequency (RF).

Spectroscopic methods have proved to be the most widely used tools for the elucidation of molecular structure as well as the quantitative and qualitative determination of both inorganic and organic compounds.

This review gives a short introduction to the four widely used Material Characterization techniques. Auger Electron Spectroscopy; a tool used for study of surfaces; this technique is commonly used for probing chemical and compositional surface environments. Field Ion Microscopy has been used to study the behavior of surfaces and the behavior of adatoms on surfaces. Low Energy Electron Diffraction is used for finding the surface structure of crystalline materials by collision with a low energy electron beam and observing the diffracted electrons as spots on fluorescent screen. Raman Spectroscopy is a technique used to examine low frequency modes in a system which helps in the determination of temperature and the crystallographic orientation of a sample. Following Section define the basic principle, instrumentation, applications and limitations of these characterization techniques.

II. DESCRIPTION OF TECHNIQUE

(A) AUGER ELECTRON SPECTROSCOPY (AES)

Auger Electron Spectroscopy provides information about the chemical composition of the outermost material comprising a solid surface. The advantages of AES over other surface analysis methods are better spatial resolution, surface sensitivity and detection of lighter elements. Detection limits for elements range from about 0.01 to 0.1 at%. AES uses a primary electron beam to excite the sample surface. When an electron in the inner shell is ejected from a sample atom by the bombardment with a primary electron, an electron from an outer shell fills the hole. To compensate the energy change from this transition, an Auger electron is emitted. For light elements, the probability is maximum for the emission of an Auger electron, which accounts for the light-element sensitivity for this technique [2]. The energy of the emitted Auger electron is feature of the element from which it was came from. Detection and energy analysis of the emitted Auger electrons produces a spectrum of Auger electron energy versus the relative abundance of electrons. Peaks in the spectrum identify the elemental composition of the sample surface. In some cases, the chemical state of the surface atoms can also be determined from energy shifts and peak shapes [2].

(i) Principle

AES uses electrons as primary radiation. The analyzed electrons are Auger electrons that are ejected as an outcome of the return of the ionized atom to its ground state.
Fig. 1. Shows a schematic representation of the processes involved in the Emission of an Auger electron.

For the example shown in Figure 1, a hole is created on the K level in the initial ionization step. This requires a primary energy greater than the binding energy of the electron in that shell. For the ionization to be efficient, a primary energy of about 5 times the binding energy is taken. In practice, typical primary energies are 5 and 10 keV. The hole is created by either the primary beam, or the backscattered secondary electrons the atom relaxes by filling the hole with an electron coming from an outer level, shown as L1. As a result, the energy difference $E_K - E_{L1}$ becomes available as excess energy, which can be used in two ways. The emission of an X-ray at that energy may occur or the energy may be given to another electron, either in the same level or in a shallower one, as is the case in the example, to be ejected. The first of the two competing processes is X-ray fluorescence, the second Auger emission. The probability for Auger emission is much higher for core levels with binding energies below about 2 keV [3].

Auger electrons are emitted in the relaxation of an excited ion with an inner shell vacancy. In this process an electron from a higher energy level fills the inner shell vacancy with the simultaneous emission of an Auger electron. This simultaneous two electron rearrangement results in a final state with two vacancies. Auger electron emission is one of two relaxation mechanisms possible in an excited ion. The other is X-ray fluorescence, in which a photon is emitted. The two relaxation processes for an excited ion are shown in the energy level diagrams of Figure 1. [3]

Auger electron emission is the more probable decay mechanism for low energy transitions, i.e., for low atomic number elements with an initial vacancy in the K shell and for all elements with initial vacancies in the L or M shells. By choosing an appropriate Auger transition, all elements (except H and He) can be detected with high sensitivity. Auger transitions are typically labeled by the energy levels of the electrons involved, using X-ray spectroscopy nomenclature. The first label corresponds to the energy level of the initial core hole. The second and third labels refer to the initial energy levels of the two electrons involved in the Auger transition. Thus the Auger transition shown in Figure 1 is a KLLIII transition, or simply a KL transition.

(ii) Instrumentation

Figure 2 represents the principal components of a complete Auger microscope. These include:

- An electron optical column for generating, focusing and scanning the incident electron beam. Typically, beam energy will be variable over a range of (0–25) keV, beam current over (1–100) nA, and beam diameter varying over (10–100) nanometers.
- A secondary electron detector (SED) for doing simple secondary electron imaging of the sample.
- A CMA or CHA electron spectrometer with multichannel detection.
- A scanning argon ion gun for sample cleaning and sputter depth profiling.
- A UHV analysis chamber (base pressure 5 ~10 Torr) with a load locked sample introduction system.
- A sample positioning system (sample stage), typically with five axes of motorized motion . . . X, Y, Z, tilt and rotation.
- Necessary electronics for digital control of all instrument functions and data acquisition.
- An instrument computer with sophisticated instrument control, data acquisition, data analysis, and data presentation software.
- A display for live and stored images, spectra and profiles.

An electron beam is focused onto a specimen and emitted electrons are deflected around the electron gun and pass through an aperture towards the back of the CMA. These electrons are then directed into the electron multiplier for analysis. Varying voltage at the sweep supply allows derivative mode plotting. An ion gun can be used for depth profiling experiments [3]

(iii) Limitations:

There are various sample constraints and potential artifacts that may limit the applicability of AES for some materials or distort the interpretation of the Auger data [4]:
Samples must be UHV compatible, and of appropriate size to fit into an Auger system.

Insulators may charge-up under the electron beam, causing an energy shift in the Auger spectrum. In extreme cases, insulators may charge to the point that no meaningful spectra can be collected.

The surface composition of some samples may be changed by electron stimulated desorption, adsorption, diffusion, dissociation, oxidation, or reduction. These effects may be observed by a time dependence of the surface composition, and may be minimized by reducing the primary beam current density and total electron dose. Some materials, such as organics, can be severely decomposed by the electron beam.

Differences in sputter yield for elements on the surface, and other ion beam induced damage may change the surface composition during ion sputtering.

For some analytical geometry, a rough surface topography may result in analytical shadowing of portions of the surface from the electron beam, the ion beam or the spectrometer.

(iii) Applications
Auger electron spectroscopy is a very powerful surface analytical technique that has found applications in many fields of solid-state physics and chemistry [4]. AES is used to monitor the elemental composition of surfaces during physical property measurements. Several phenomena such as adsorption, desorption, surface segregation from the bulk, measurement of diffusion coefficients, and catalytic activity of surfaces have been investigated using AES. It has also been used to study the surface compositional changes in alloys during ion sputtering. Chemical properties such as corrosion, stress corrosion, oxidation, and catalytic activity and mechanical properties such as fatigue, wear, adhesion, resistance to deformation processes, and surface cracking depend on surface properties. Similarly, grain boundary chemistry influences mechanical properties such as low- and high-temperature ductility and fatigue, chemical properties such as inter-granular corrosion and stress corrosion, and electrical properties. AES has been used to relate surface and grain boundary chemistry to properties of materials. AES has proved to be extremely valuable compared to most other techniques, which are limited by either large sampling depth or poor sensitivity [4].

B. FIELD ION MICROSCOPY (FIM)

The field-ion microscope (FIM) is a unique analytical instrument that can analyze metals and semiconducting materials on the atomic scale [5]. In recent years, it has developed into one of the most powerful instruments available for routine micro-structural and micro-chemical analysis of materials. The types of investigations that have been performed included many metallurgical subjects including phase transformations, segregation, diffusion, catalysis, and radiation damage.

(i) Principle
FIM relies on the electric-field-induced ionization of inert gas atoms in the vicinity of a charged surface. When a very sharp metallic needle is subjected to a high voltage of a few kilovolts, an intense electric field is generated at the surface. This electric field is generated by the positive charges present at the surface. Indeed, the application of the high voltage induces the free electrons to be, on average, displaced inwards by a small amount to screen the electric field, leaving partly charged atoms at the very surface [6].

Since the electric field at the surface is directly proportional to the charge density, it is higher around these local protrusions. In the case of an atomically smooth curved surface, these protrusions correspond to the edges of atomic terraces. By imaging the distribution of the field intensity at the surface, the field ion microscope provides an atomically resolved image of the surface itself [6].

(ii) Instrumentation
Figure 3 shows the key components of instrumentation for FIM. The instrument consists of an ultra-high vacuum chamber, which is required to operate at a base pressure below 10–8 Pa (~10–10 Torr). This is achieved by using high compression rate turbo-molecular pumps backed by rotary pumps. Low specimen temperatures must be maintained to achieve high resolution. Early microscope designs simply used liquid nitrogen, hydrogen or helium to cool the specimen [5]. A closed-cycle helium cryostats is generally used to reach temperatures below 20 K. A high-voltage DC power supply is connected to the specimen in order to generate the required electric field. Once the tip is positioned and thermally stabilized, a very low pressure, in the range of 10–3 to 10–4 Pa (~ 10 DC–5 to 10–6 Torr), of imaging gas is introduced into the chamber. The most commonly used gases are He and Ne, although in some cases H2 has also been used. Multiple gases in combination can also be used to resolve images from several phases at the same time [5, 6].

The FIM screen is composed of a stack of biased micro-channel plates (MCPs), which is used as an image intensifier, placed immediately in front of a phosphor screen. A MCP consists of an array of tiny glass tubes with a diameter of the order of a few tens of microns, covered by a thin conductive layer [7]. The tubes are oriented at an angle of approximately 15–5 to normal. Generally, one of the faces of the MCP is biased at about –1 kV, creating a high surface charge density. The opposite side is grounded, which induces an electric field between the two faces. When a particle, ion, electron or photon hits the inside wall of one of the tubes, secondary electrons are emitted by the surface. By virtue of the electric field, they are driven towards the back face. Each time one of these electrons collides with the wall of the tube, another cluster of secondary electrons is ejected, ultimately producing a cascade of several thousand electrons for each corresponding ion that impacts the surface. The electrons are consequently focused onto the phosphor by an electric field imposed between the MCP and the phosphor screen, which produces a spot of light on impact of the electrons. The detection-efficiency of the MCP can be defined as the probability that it will successfully detect a single ion striking its surface. This
detection-efficiency is limited by the open area of the MCP, i.e. the fraction of the surface area of the detector that serves as an opening to the micro-channels. This fraction has an approximate value of between 50% and 60%. However, in the case of FIM, high number of imaging-gas ions contributes to form a single spot within the image. Indeed even if 50% of the ions reaching the MCP are not detected, there will still be thousands of ions that are successfully detected and will contribute to the formation of each spot. Hence, the detection-efficiency limitation imposed by the MCPs is not a significant issue in FIM [7].

(iii) Limitations
Application of FIM is limited by the materials which can be fabricated in the shape of a sharp tip, can be used in an ultra high vacuum (UHV) environment, and can tolerate the high electrostatic fields. For these reasons, refractory metals with high melting temperature (for e.g. W, Mo, Pt, Ir) are conventional objects for FIM experiments. Metal tips for FIM are prepared by electro-polishing (electrochemical polishing) of thin wires. However, these tips usually contain many asperities. The final preparation procedure involves the in situ removal of these asperities by field evaporation just by raising the tip voltage. Field evaporation is a field induced process which involves the removal of atoms from the surface itself at very high field strengths and typically occurs in the range 2-5 V/Å. The effect of the field in this case is to reduce the effective binding energy of the atom to the surface and to give, in effect, a greatly increased evaporation rate relative to that expected at that temperature at zero fields. This process is self-regulating since the atoms that are at positions of high local curvature, such as adatoms or ledge atoms, are removed preferentially. The tips used in FIM is sharper (tip radius is 100–300 Å) compared to those used in FEM experiments (tip radius ~1000 Å) [7].

(iv) Applications
FIM has been used to study dynamical behavior of surfaces and the behavior of adatoms on surfaces. The problems studied include adsorption desorption phenomena, surface diffusion of adatoms and clusters, adatom-adatom interactions, step motion, equilibrium crystal shape, etc [6]. However there is the possibility of the results being affected by the limited surface area (i.e. edge effects) and by the presence of large electric field.

(C) LOW ENERGY ELECTRON DIFFRACTION

Low Energy Electron Diffraction (LEED) is one of the most successful surface science techniques for structure determination ever invented. For more than 50 years it has been the dominant method to study the structure and morphology of two dimensional plane surfaces. A vast variety of techniques and extremely sophisticated applications have been developed till date. For the structure analysis of atomic positions in the surface unit cell, LEED analysis has been developed with an accuracy of less than a few hundred Å. Moreover, the determination of complex surface structures has been automated [8].

Low-energy electron diffraction (LEED) is used for the determination of the surface structure of crystalline materials by bombardment with a collimated beam of low energy electrons (20–200 eV) and observation of diffracted electrons as spots on a fluorescent screen [8].

Fig 3: Instrumentation for FIM

LEED may be used in 2 ways;

1. Qualitatively, where the diffraction pattern is recorded and analysis of the spot positions gives information on the symmetry of the surface structure. In the presence of an adsorbate the qualitative analysis may reveal information about the size and rotational alignment of the adsorbate unit cell with respect to the substrate unit cell.

2. Quantitatively, where the intensities of diffracted beams are recorded as a function of incident electron beam energy to generate the so-called I-V curves. By comparison with theoretical curves, these may provide accurate information on atomic positions on the surface at hand.

(i) Principle
The basic principle of a standard LEED experiment is very simple: a collimated mono-energetic beam of electrons is directed towards a single crystal surface and the diffraction
pattern of the elastically back-scattered electrons is recorded using a position-sensitive detector [8].

By the principles of wave-particle duality, the beam of electrons may be equally regarded as a succession of electron waves incident normally on the sample. These waves will be scattered by regions of high localized electron density, i.e. the surface atoms, which can therefore be considered to act as point scatterers. The wavelength of the electrons is given be the de Broglie relation [9]:

\[ \lambda = \frac{h}{p} \] (where \( p \) - electron momentum)

Now,

\[ p = m.v = (2mE_k)^{1/2} = (2m.e.V)^{1/2} \]

where

- \( m \) - mass of electron [ kg ]
- \( v \) - velocity [ m s \(^{-1} \) ]
- \( E_k \) - kinetic energy
- \( e \) - electronic charge
- \( V \) - acceleration voltage (= energy in eV)

Wavelength, \( \lambda = h / (2m.e.V)^{1/2} \)

(Where \( h = 6.62 \times 10^{-34} \) J s, \( e = 1.60 \times 10^{-19} \) C, \( m_e = 9.11 \times 10^{-31} \) kg). The range of wavelengths of electrons employed in LEED experiments is seen to be comparable with atomic spacing, which is the necessary condition for diffraction effects associated with atomic structure to be observed. Consider, first, a one dimensional (1-D) chain of atoms (with atomic separation \( a \)) with the electron beam incident at right angles to the chain. This is the simplest possible model for the scattering of electrons by the atoms in the topmost layer of a solid; in which case the diagram below would be representing the solid in cross-section with the electron beam incident normal to the surface from the vacuum above.

\[ d = a \sin \theta = n \lambda \]

where:

- \( \lambda \) - wavelength
- \( n \) - integer (\(-1, 0, 1, 2, \ldots\) )

For two isolated scattering centres the diffracted intensity varies slowly between zero (complete destructive interference; \( d = (n + \frac{1}{2}) \lambda \)) and its maximum value (complete constructive interference; \( d = n \lambda \)) - with a large periodic array of scatterers, however, the diffracted intensity is only significant when the "Bragg condition"

\[ a \sin \theta = n \lambda \]

is satisfied exactly. The Fig 6 shows a typical intensity profile for this case.

Consider the LEED pattern from an fcc(110) surface. In the diagram below the surface atomic structure is shown on the left, as if you are viewing it from the position of the electron beam.

**Fig 5: Solid Interacted by an low energy electron beam**

Considering the backscattering of a wavefront from two adjacent atoms at a well-defined angle, \( \theta \), to the surface normal then it is clear that there is a "path difference" \( (d) \) in the distance the radiation has to travel from the scattering centres to a distant detector (which is effectively at infinity) -
gun in the LEED experiment. The primary electron beam would then be incident normally on this surface as if fired from your current viewpoint and the diffracted beams would be scattered from the surface back towards yourself. The diffraction pattern on the right illustrates how these diffracted beams would impact upon the fluorescent screen.

A much better method of looking at LEED diffraction patterns involves using the concept of reciprocal space. It can be readily shown that the observed LEED pattern is a (scaled) representation of the reciprocal net of the pseudo-2D surface structure. The reciprocal net is determined by (defined by) the reciprocal vectors: $a_1^*$ & $a_2^*$ (for the substrate) and $b_1^*$ & $b_2^*$ (for the adsorbate). Initially consider just the substrate. The reciprocal vectors are related to the real space unit cell vectors by the scalar product relations:

$$a_1 \cdot a_2^* = a_1^* \cdot a_2 = 0$$

$$a_1 \cdot a_1^* = a_2 \cdot a_2^* = 1$$

when $A = 0$ degrees ($\cos A = 1$) this simplifies to a simple reciprocal relationship between the lengths of $a_1$ and $a_1^*$ (and $a_2$ and $a_2^*$) of the form : $|a_1| = 1 / (|a_1^*| \cos A)$ , where $A$ is the angle between the vectors $a_1$ and $a_1^*$. 

By contrast, the diagram below shows the diffraction pattern that might be expected if the energy of the incident electrons is doubled - some of the second order spots are now visible and the pattern as a whole has apparently contracted in towards the central (00) spot.

(ii) Instrumentation

The standard modern LEED system is of the “rear view” type, which is schematically depicted in Figure. The incident electron beam, accelerated by the potential, is emitted from the electron gun behind a transparent hemispherical fluorescent screen and hits the sample through a hole in the screen. Typically, the electron beam has a current of around 1 $\mu$A and a diameter of 0.5 to 1 mm. The surface is in the centre of the hemisphere so that all back-diffracted electrons travel towards the LEED screen on radial trajectories.

Before the electrons hit the screen they have to pass a retarding field energy analyzer (RFA). It consists of four (sometimes three) hemispherical grids concentric with the screen, each containing a central hole, through which the electron gun is inserted. The first grid (nearest to the sample) is connected to earth ground as is the sample, in order to provide a field-free region between the sample and this grid. A negative potential is applied to the second and third grid, the so-called suppressor grids. These repel all electrons that have undergone non-elastic scattering processes and have lost more than eV(typically around 5eV) of their original kinetic energy.
(iii) Limitations

Low Electron Energy Diffraction method is limited due to the following constraints [10]:

- It involves sophisticated multiple scattering calculations during quantitative surface structure determination.
- Difficult to distinguish when two domains of different structures coexist.
- Sensitive molecular adsorbates cause electron beam damage.

(iv) Applications

LEED has found its major applications in the study of determination of surface of crystalline material because of the following merits [10]:

- Surface order and cleanliness
- Surface atomic structure
- Step density – get step height/density from angular beam profile.
- Phase transition in over layers - structure may undergo transition with change in coverage.
- Dynamics of ordering, disordered, growth, phase transitions

(D) RAMAN SPECTROSCOPY

Raman Spectroscopy is a form of vibrational spectroscopy, which involves emission and absorption of infrared (IR) and visible light (as the form of light-based interaction with the molecule). Specifically, Raman spectroscopy is concerned with the absorption and emission of visible light; moreover, unlike Rayleigh scattering (the frequency invariant phenomenon which makes the sky blue), Raman scattering requires a change in frequency; hence, it is actually the inelastic scattering of light [11].

(i) Principle

The Raman effect is based on molecular deformations in electric field E determined by molecular polarizability. The laser beam can be considered as an oscillating electromagnetic wave with electric vector E. Upon interaction with the sample it induces electric dipole moment which deforms molecules. Because of periodical deformation, molecules start vibrating with characteristic frequency. Amplitude of vibration is called a nuclear displacement. In other words, monochromatic laser light with frequency excites molecules and transforms them into oscillating dipoles. Such oscillating dipoles emit light of three different frequencies when:

1. A molecule with no Raman-active modes absorbs a photon with the frequency. The excited molecule returns back to the same basic vibrational state and emits light with the same frequency as an excitation source. This type if interaction is called an elastic Rayleigh scattering.
2. A photon with frequency is absorbed by Raman active molecule which at the time of interaction is in the basic vibrational state. Part of the photon’s energy is transferred to the Raman-active mode with frequency and the resulting frequency of scattered light is reduced. This Raman frequency is called Stokes frequency, or just “Stokes”.
3. A photon with frequency is absorbed by a Raman-active molecule, which, at the time of interaction, is already in the excited vibrational state. Excessive energy of excited Raman active mode is released, molecule returns to the basic vibrational state and the resulting frequency of scattered light goes up. This Raman frequency is called Anti-Stokes frequency, or just “Anti-Stokes”.

![Fig 9: Schematic of LEED Set Up](image)
Note the difference between Stokes, Anti-Stokes, and Rayleigh scattering as seen in Figure 10.

![Spectra of the scattered radiation obtained when a sample of CCl4 was interacted with a laser beam having a wavelength of 488.0 nm.](image)

Fig 10: Spectra of the scattered radiation obtained when a sample of CCl4 was interacted with a laser beam having a wavelength of 488.0 nm.

About 99.999% of all incident photons in spontaneous Raman undergo elastic Rayleigh scattering. This type of signal is useless for practical purposes of molecular characterization. Only about 0.001% of the incident light produces inelastic. Spontaneous Raman scattering is very weak and special measures should be taken to distinguish it from the predominant Rayleigh scattering. Instruments such as notch filters, tunable filters, laser stop apertures, double and triple spectrometric systems are used to reduce Rayleigh scattering and obtain high-quality Raman spectra [11].

(ii) **Instrumentation**

A Raman system typically consists of four major components:

1. Excitation source (Laser).
2. Sample illumination system and light collection optics.
3. Wavelength selector (Filter or Spectrophotometer).
4. Detector (Photodiode array, CCD or PMT).

A sample is normally illuminated with a laser beam in the ultraviolet (UV), visible (Vis) or near infrared (NIR) range. Scattered light is collected with a lens and is sent through interference filter or spectrophotometer to obtain Raman spectrum of a sample [12].

Since spontaneous Raman scattering is very weak the main difficulty of Raman spectroscopy is separating it from the intense Rayleigh scattering. More precisely, the major problem here is not the Rayleigh scattering itself, but the fact that the intensity of stray light from the Rayleigh scattering may greatly exceed the intensity of the useful Raman signal in the close proximity to the laser wavelength. In many cases the problem is resolved by simply cutting off the spectral range close to the laser line where the stray light has the most prominent effect. People use commercially available interference (notch) filters which cut-off spectral range of ± 80-120 cm⁻¹ from the laser line. This method is efficient in stray light elimination but it does not allow detection of low-frequency Raman modes in the range below 100 cm⁻¹ [12].

Stray light is generated in the spectrometer mainly upon light dispersion on gratings and strongly depends on grating quality. Raman spectrometers typically use holographic gratings which normally have much less manufacturing defects in their structure than the ruled ones. Stray light produced by holographic gratings is about an order of magnitude less intense than from ruled gratings of the same groove density. Using multiple dispersion stages is another way of stray light reduction. Double and triple spectrometers allow taking Raman spectra without use of notch filters. In such systems Raman-active modes with frequencies as low as 3-5 cm⁻¹ can be efficiently detected. In earlier times people primarily used single-point detectors such as photon-counting Photomultiplier Tubes (PMT). However, a single Raman spectrum obtained with a PMT detector in wave number scanning mode was taking substantial period of time, slowing down any research or industrial activity based on Raman analytical technique. These days, more and more often researchers use multi-channel detectors like Photodiode Arrays (PDA) or, more commonly, a Charge-Coupled Devices (CCD) to detect the Raman scattered light. Sensitivity and performance of modern CCD detectors are rapidly improving. In many cases CCD is becoming the detector of choice for Raman spectroscopy [12].

(iii) **Limitations**

There are certain limitations associated with Raman Spectroscopy which are listed below [13, 14]:

- It cannot be used for metals or alloys.
- The Raman Effect is very weak. The detection needs a sensitive and highly optimized instrumentation
- Fluorescence of impurities or of the sample itself can hide the Raman spectrum
- Sample heating through the intense laser radiation can destroy the sample or cover the Raman spectrum

(iv) **Applications:**

Raman Spectroscopy is an ideal tool for characterization of graphene, providing comprehensive information about the structure and layer thickness of graphene samples. It is highly useful for quantitative and qualitative analysis of inorganic, organic and biological samples because of the following advantages [13, 14];
• It Can be used with solids and liquids
• Sample preparation is not required
• It does not interfere by water
• It is non-destructive
• Raman spectra are acquired quickly within seconds
• Samples can be analyzed through glass or a polymer packaging
• Laser light and Raman scattered light can be transmitted by optical fibers over long distances for remote analysis
• Raman spectra can be collected from a very small volume (< 1 µm in diameter)
• Inorganic materials are normally easier analyzed by Raman than by infrared spectroscopy

(III) CONCLUSION

Most powerful and widely used experimental techniques of material characterization i.e. Auger Electron Spectroscopy, Field Ion Microscopy, Low Energy Electron Diffraction and Raman Spectroscopy were briefly discussed in this paper. The merits and the associated limitations along with applications with the respective analytical technique were also highlighted.

REFERENCES